

# Synthesis and Characterization of Polymeric Composite Films by Designed Extruders

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**Abstract**— Polypropylene (PP) is extensively used in many industrial areas such as the automobile, household goods, packaging and electronic appliance properties applications due to its good performance and processing properties as well as low cost. Diatomite is a siliceous sedimentary rock. It has a unique combination of physical and chemical properties such as high porosity, high permeability, small particle size, large surface area and low thermal conductivity. For this purpose, in this study, the preparation of composite materials with good properties from a mixture of polypropylene and diatomite was considered. The surface of diatomite particles was treated by benzyltrimethylammonium chloride (BTMAC) and the diatomite filled polypropylene (PP) composites with a weight percentage range of the filler from 0 to 30% were prepared by means of melt blending in a designed single-screw extruder. The characterizations of the composite films synthesized by the laboratory type extruder were performed with FTIR, TGA, and SEM devices. Tensile strength tests were also performed for the mechanical properties of the synthesized films.

**Index Terms** — Characterization, Composite, Diatomite, Modification, Polymer, Extruder

## I. INTRODUCTION

Polypropylene is one of the most commonly used polymers in many sectors such as automobile, household goods, packaging and electronic appliance properties applications due to its good performance and processing properties as well as low cost [1]. However, the use of polypropylene in the industry is limited under certain circumstances. Therefore, how to improve the dimensional stability and mechanical properties is extensively paid attention. For this purpose, the physical and mechanical properties of polymers are being greatly improved by filling with rigid inorganic additives, such as  $\text{CaCO}_3$  [2-5], talc [6], mica [7], kaolin [8], montmorillonite [9] and layered silicate [10].

Diatomite in its natural state is a soft rocklike material consisting essentially of the skeletal remains of a variety of singlecelled microscopic plants known as diatoms. They are generally amorphous, hydrated or opaline silica,  $\text{SiO}_2 \cdot n\text{H}_2\text{O}$ , with various amounts of impurities such as silica sand, clay minerals, metal salts and organic matter. Diatomite is a low-cost, environment-friendly and natural micro/nanostructured material derived from sedimentary silica, and has cylindrical and plate morphologies with well-developed mesoporous/macroporous structures [11].

In this study, the diatomite was modified with benzyltrimethylammonium chloride (BTMAC) and mixed with polypropylene at different mass percentages. Thin films

were synthesized with the prepared mixtures by using laboratory type of extrusion machines. Meanwhile, the laboratory type of extrusion machines used for the preparation of composite films have been designed and manufactured by me. Characterization of the synthesized films was performed using FTIR, SEM, TG and mechanical test devices.

## II. MATERIALS AND METHODS

### Materials

Polypropylene (PP) used as a matrix material in the synthesis of composite film is supplied from PETKİM Co., Turkey. The diatomite used as a filler material is of analytical purity and is obtained from commercially sold companies. The particle size of the diatomite was reached to 45-109  $\mu\text{m}$  by grinding and sieving. Benzyltrimethylammonium chloride (BTMAC) was purchased from Sigma-Aldrich and its molecular structure was given in Figure 1.

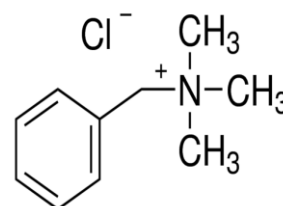


Figure 1. Molecular structure of BTMAC

### Modification of Diatomite with BTMAC

For the modification of diatomite, 250 mL of a 0.1 M BTMAC solution was shaken with 5 g of diatomite in a shaking bath at constant temperature for 2.5 hours. The modified diatomite was removed from suspension by centrifugation and dried in an oven at 45 °C for 1 day.

### Preparation of composites

Before mixing, PP and diatomite samples were dried at 90 °C for 4 h. and then cooled to room temperature in a desiccator. After mixing at different mass percentages (0-30%), the PP and diatomite samples were combined in a single-screw extrusion apparatus. The mixture was carried out at a temperature range of 170-220 °C and a screw speed of 20 rpm.

The laboratory type extrusion devices used for preparing composite films are shown in Figure 2 and 3. The parts of designed microprocessor controlled panel are shown in Figure 4.

Design and manufacture of these original laboratory-type-extruders shown below were made by me.

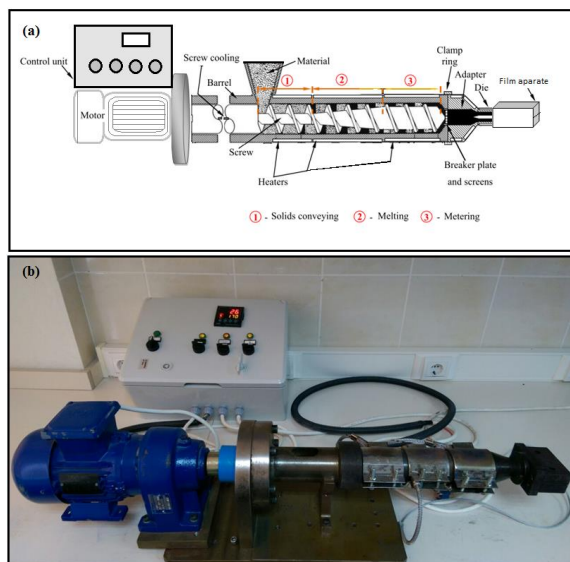


Figure 2. (a) Schematic and (b) original device representation of a laboratory-type-extruder used in the preparation of polypropylene composite films.

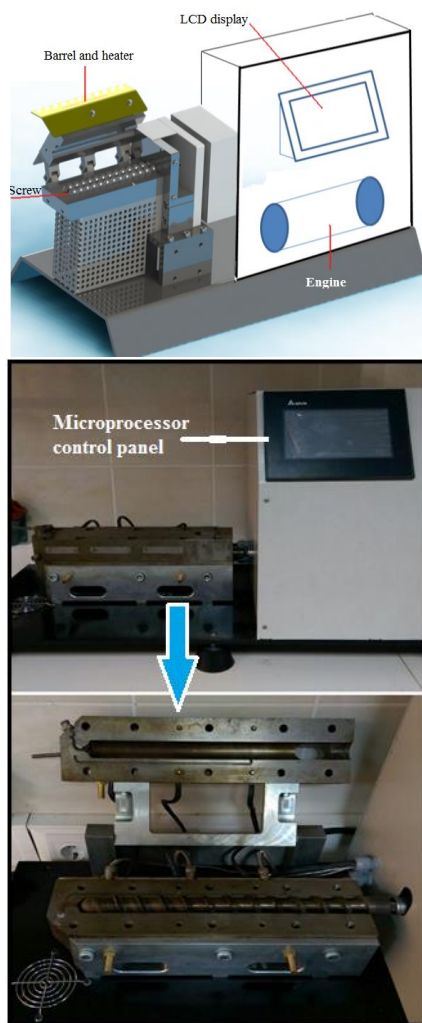


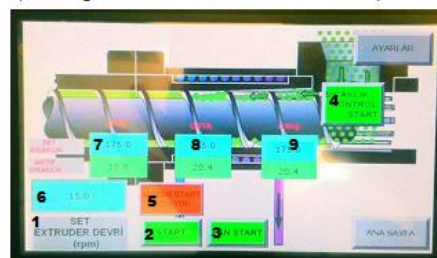
Figure 3. Microprocessor controlled second original laboratory-type-extruder (Schematic and original device representation).

## Characterization and Tests

The characterizations of the composite films synthesized

by the laboratory type extruders were performed with FTIR, TG, and SEM devices. The morphologies of samples were observed in scanning electron microscopy (SEM) of Hitachi. Fourier transform infrared (FT-IR) spectra were recorded on a Perkin Elmer FTIR spectrometer using ATR device. The Thermo-Gravimetric (TG) analysis was obtained simultaneously using a Perkin Elmer instrument. Tensile strength tests were also performed for the mechanical properties of the synthesized films. A mechanical test was conducted according to the ASTM standard.

Designed LCD Display (Real screenshot) (Microprocessor with Touchscreen)



1. Screw rotation speed setting
2. Heater start button
3. Ventilation start button
4. Temperature control
5. Start the engine
6. Current screw rotation speed
7. Front zone heater setting
8. Center zone heater setting
9. End zone heater setting

Figure 4. The parts of designed microprocessor controlled panel.

## III. RESULTS AND DISCUSSION

### FTIR Spectra

Figure 4 shows the FTIR spectra of polypropylene (PP), diatomite (D), BTMAC and their composite films. • As seen in Figure 4, spectrum of PP, values of 1455.73, 1375.60, and 1167.22  $\text{cm}^{-1}$  are  $-\text{CH}_2$  bending,  $-\text{CH}_3$  bending, and C-C stretching, respectively. From Figure 4, spectrum of diatomite, the band at  $\sim 1634 \text{ cm}^{-1}$  is due to OH bending vibrations of adsorbed water in sheet silicate minerals. The  $\sim 1023 \text{ cm}^{-1}$  band arises from the Si-O-Si vibration. The  $\sim 795 \text{ cm}^{-1}$  band occurs because of the OH translational vibration [12] The characteristic peaks in the BTMAC spectrum are the aromatic C-H stretching at 3006.40  $\text{cm}^{-1}$ , the C-H stretching in  $\text{N}^+-\text{CH}_3$  at 2900  $\text{cm}^{-1}$ , the C = C stretching at 1493.55  $\text{cm}^{-1}$  and the C- $\text{N}^+$  stretches at 739.44  $\text{cm}^{-1}$ . The BTMAC modified diatomite (MD) spectrum has characteristic peaks both BTMAC and diatomite. This spectrum may show that MTMAC interacts with diatomite.

The peaks of the FTIR spectra of the D/PP composite films prepared from 1% to 30% by weight (w/w) are  $-\text{CH}_2$  and  $-\text{CH}_3$  bending at about 1455 and 1375  $\text{cm}^{-1}$ , respectively, Si-O-Si vibrations of diatomite were observed at 1030.27  $\text{cm}^{-1}$ . As the amount of D in the D / PP composites increases, the peaks of the Si-O-Si vibrations (in the range of 1018-1045  $\text{cm}^{-1}$ ) naturally become more apparent in the FTIR spectra. The

spectrum of MD/PP composite film is similar to that of D/PP (ratio of 30/70) composite film. However, in the MD/PP spectrum, characteristic peaks of BTMAC were observed in the range of 650-750  $\text{cm}^{-1}$ . This result indicates the presence of interaction between MD and PP in the composite films.

### Thermal Properties

Thermogravimetric analysis (TGA) was used to study the thermal stability of the samples. Figure 5 shows the weight loss temperature curves (TG) of PP and containing either D or MD. According to the thermal behavior observed for these composites the decomposition mechanism could be attributed principally to the functionalization process of PP occurring in one step located around 400-550°C. The polar organic compound present in modifier and the MD could be the principal decomposition events to explain the thermal stability of composites. In the case of PP the thermal degradation is known to occur principally by a random scission mechanism [13].

waves. These small waves will absorb relevant impact deformation energy or fracture energy, leading to improving obviously the impact fracture toughness of the composite systems.

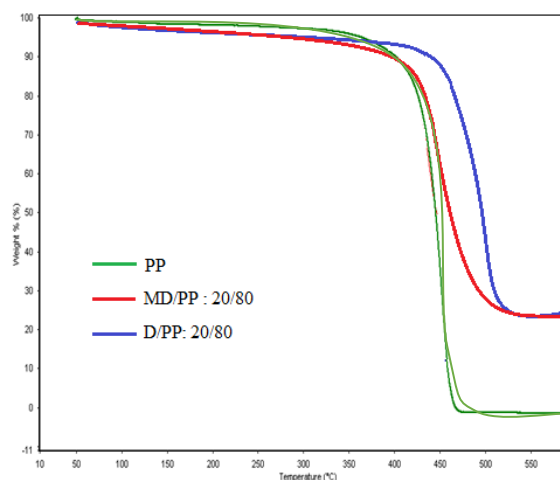


Figure 5. Thermogravimetric analysis of PP and selected their composite films

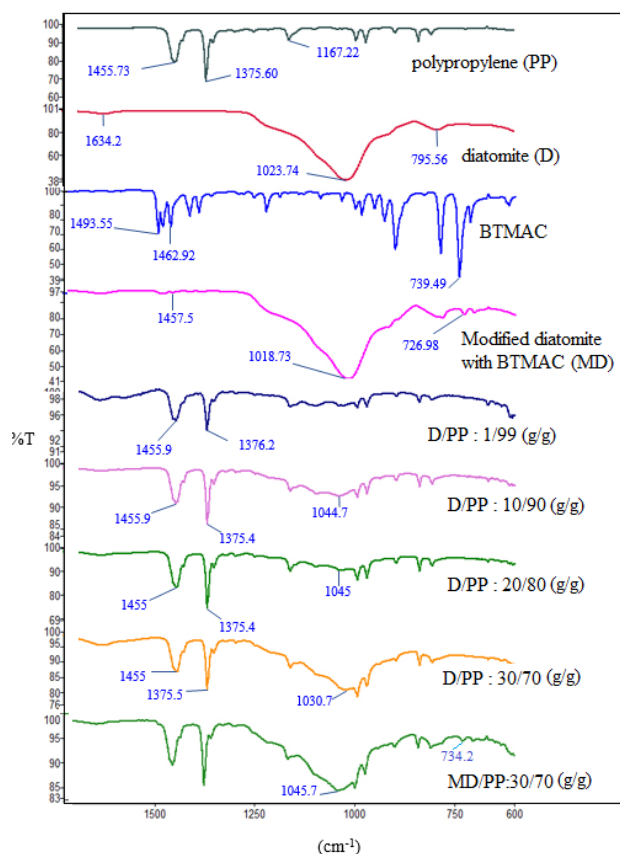


Figure 4. FTIR spectra of PP, D, BTMAC, and composite films

### Morphological analysis

Figure 6 displays respectively the SEM images of PP, D/PP, MD/PP systems with different mass fraction of the fillers. There are two fracture modes in polymeric composites: ductile fracture and brittle fracture. The neat PP belongs to a typical brittle fracture, where the fracture section was flat [14]. In Figure 6, the SEM image of neat polypropylene film looks smooth. The surfaces of D/PP and 1% of MD/PP composite films are rough and brittle. However, the SEM image of a 2.5% MD/PP composite films looks like small

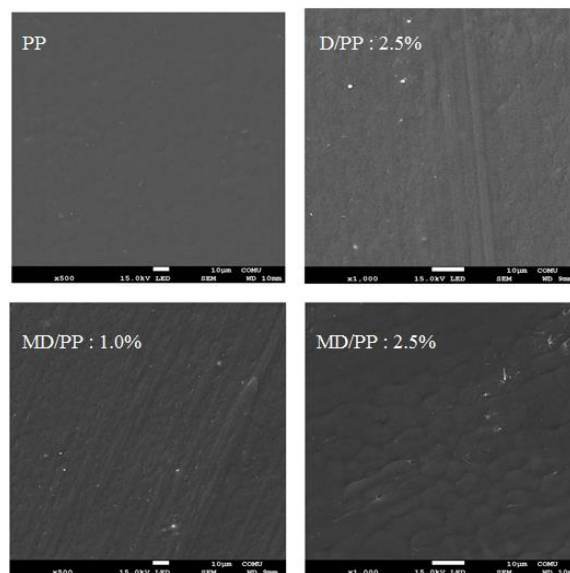


Figure 6. The SEM images of samples

### Mechanical Properties

In this study, the variation of the mass percentages of the filler materials (D or MD) versus the tensile strengths of the PP, D / PP and MD / PP composite films synthesized is shown in Figure 7 and 8. Figure 7 shows the variation of tensile strengths with different mass ratio of the D/PP composite films.

As seen Figure 7, the tensile strength of neat PP was tested as 17.58 MPa. When the two fillers were placed in the PP matrix, the tensile strength of the samples changed in almost the same ratio. The tensile strength of MD-filled composites at lower percentages is slightly greater than that of D-filled composites. The reason of this, the poor adhesion between D filler and the polymer may be the major reason for the poor



tensile strength performance and created weak interfacial regions, resulting in debonding and frictional pullout. However, in low percentages, especially 2.5%, the maximum tensile strength was tested for the composite film between BTMAC modified diatomite (MD) and polypropylene (see Figure 7b). We can explain the reason for this situation by having stronger interactions between MD and polymer. The decrease in the polymer matrix content in the composite as a function of the filler content was also responsible for the low tensile strength.

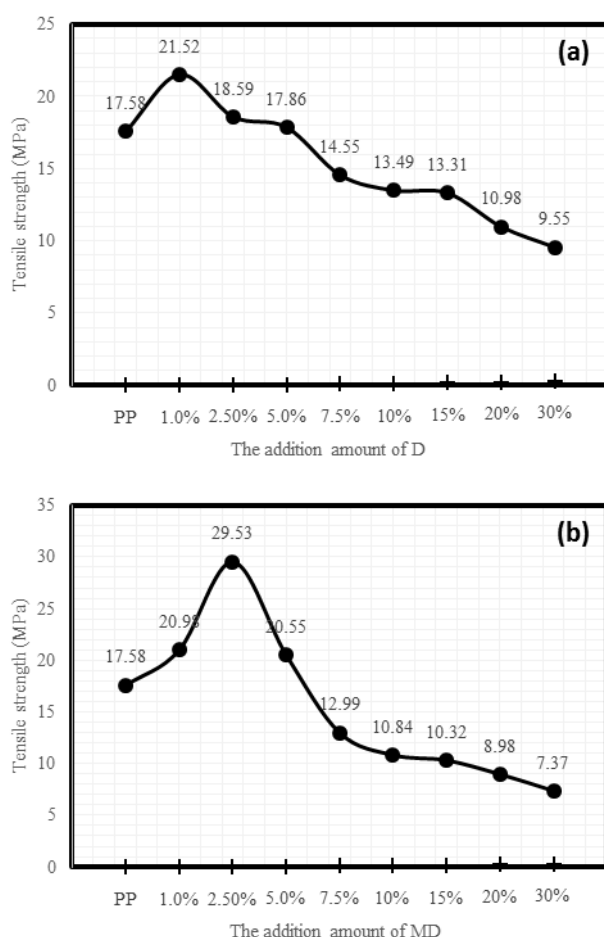


Figure 7. Tensile strength of PP, D/PP and MD/PP samples

#### IV. CONCLUSIONS

In this study, polypropylene (PP) used as a matrix material in the synthesis of composite film. The diatomite was modified with benzyltrimethylammoniumchloride (BTMAC) and mixed with polypropylene at different mass percentages. Thin films were synthesized with the prepared mixtures by using laboratory type of extrusion machines. Characterization of the synthesized films was performed using FTIR, SEM, TG and mechanical test devices. FTIR spectra show that diatomite is modified with BTMAC, polypropylene is interacting with diatomite and MD. Thermal analyzes indicate that the thermal stability of the composite specimens increases at least. SEM images showed that the surface morphology of the composite films changed. Finally, from the mechanical test results, the highest tensile strength was found to be 29.53 MPa for MD films at 2.5% (w/w). As a result, diatomite and

BTMAC modified diatomite fillers were shown to be usable with polypropylene matrices.

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